AU9481809

(12) PATENT ABRIDGMENT (11) Document No. AU-B-81809/94
(19) AUSTRALIAN PATENT OFFICE (10) Acceptance No. 659400

(Australian Petty Patent)

(54) Title
THERMOSETTING MOULDING COMPOSITIONS AND ASSOCIATED METHODS

International Patent Classification(s)
(51)[6] B29C 039/10 B29C 039/12 B32B 001/02 H01M 002/02

(21) Application No. : 81809/94 (22) Application Date : 23.12.94

(43) Publication Date : 11.05.95

(45) Publication Date of Granted Application : 11.05.95

(62) Related to Division(s) : 80429/94

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(57) Claim

1. A container holding acid in an electrolytic cell, the container having a laminate wall comprising an inner layer and an outer layer, the inner layer comprising a composition of vinylester resin, continuous glass rovings in the form of surface tissues providing a very fine membrane of glass having a resin richness greater than 90%, the outer layer comprising vinylester compatible resin to promote bonding with the inner layer, glass fibre, fine, and course sand and gravel.

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P/00/011
Regulation 3.2

AUSTRALIA

Patents Act 1990

COMPLETE SPECIFICATION FOR A PETTY PATENT

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Invention Title: Thermosetting Moulding Compositions and Associated Methods

Details of Associated Application No: Australian Patent Application No. 80429/94 filed 13 December 1994.

The following statement is a full description of this invention, including the best method of performing it known to me:

THIS INVENTION relates to thermosetting moulding compositions and associated methods and in particular, but not limited to moulding compositions and methods for construction of containers for holding corrosive liquids.

It is most desirable that a container used for holding corrosive liquids be leak-proof.

A typical prior art container for holding acid in an electrolytical cell is described in U.S. Patent No. 4,885,072. This container is moulded using a composition employing vinylester or polyester thermosetting resin modified by the addition of a thinning agent, inhibitors, promoters and catalyst and the balance of the composition includes dry ingredients of crystalline silica particles taken from the groups consisting of glass beads and mica flakes. Reinforcing rods are employed in the bottom of the container.

The container is formed by mixing the resin with the dry ingredients and then pouring the same continuously into an inverted mould. The mixture is then allowed to cure at room temperature providing a final container having relatively smooth inside bottom side and end wall surfaces.

This method results in a container having a wall thickness of about 2½ inches (62mm).

While the use of the resin provides a corrosive resistant container, this technology has a number of disadvantages.

A major problem with the container described in U.S. Patent No. 4,885,072 is that the container is prone to eventually leak. This is because the thermosetting process can cause hairline cracks throughout the container wall

and these hairline cracks can be the focal point for further cracking and eventually leakage.

It is an object of the present invention to alleviate the aforementioned problems of the prior art.

In one preferred form, the present invention resides in a container for holding corrosive liquids, the container having a laminated wall comprising a relatively thin corrosion resistant inner layer moulded from a thermosetting moulding composition and a further relatively thick layer adhered to the inner layer, the further layer being moulded from a thermosetting moulding composition comprising a resinous binder mixed with granular abrasive particles providing the bulk of the composition.

In another preferred form, the present invention provides a laminar structure having adjacent layers moulded from respective thermosetting moulding compositions, the layers being adhered together to eliminate transverse fracture of the structure.

In a further preferred form, the present invention resides in a method for forming a laminate from first and second thermosetting moulding compositions, one of said compositions having an abrasive media contained therein, the method including the steps of:-

(i) providing a first layer of the first composition and allowing it to commence setting to a green state;

(ii) applying a layer of the second composition to the first layer while the first composition is still capable of reacting with the second composition but being set sufficiently to provide a defined inner layer in the finished

laminate; and simultaneously

(iii) applying vibration to the layers.

The method preferably includes a mixing step for preparing the composition having the abrasive media, the mixing step involving progressively mixing the abrasive media with a resinous liquid to form a mixture, the progressive mixing involving predominantly a folding action applied to the mixture in order to limit the amount of air in the mixture.

The method typically includes a further step of controlling the exotherm of the second composition while setting, either the control typically being by application of cooling media to a mould or by controlling the amount of catalyst in the composition.

The method preferably includes the further step of moulding the first layer as a container on a mould and subsequently releasing the moulded container from the mould to form a thin corrosion resistant container before applying the second layer, the second layer providing a supporting backing layer for the container. Preferably, the container formed by the first layer is removed from the mould and placed on a moulding jig to support the container while the backing layer is being poured. The moulding jig typically includes an outer surface adapted to inhibit adherence of the container. For example, the moulding jig typically has a smooth outer surface to ease removal of the completed laminate container from the jig.

In one typical example of application of the present invention, when it is applied to a container, a first layer comprises a composition of vinylester resin, continuous fibreglass rovings and catalyst applied to a mould having an

inverted mould plug, the composition being applied as an outer layer of the mould plug, the layer being laid to about 4mm thick and being allowed to gel and subsequently released from the mould and placed on a moulding jig surrounded by mould forming walls, a second layer comprising resin, glass fibre, fine and course river sand and gravel is folded into an intimate blend and then poured into the mould formed by the first layer and walls and onto the first layer to a thickness of about 45mm to 90mm while the mould is being vibrated.

Typically, the first layer is free of abrasive media and the first composition preferably comprises by weight 60% to 80% vinylester resin with the balance being continuous glass rovings C.S.M. and woven rovings. In another embodiment, the first layer is most preferably formed using glass surface tissues to provide a layer having a very fine membrane of glass and a resin richness of above 90% with 97% being preferable.

The second layer preferably includes the abrasive media and preferably comprises by weight 10% to 20% resin, 0% to 10% glass, 10% to 20% sand and 30% to 60% gravel with sufficient catalyst to provide about a one hour gel time.

Most preferably, the exotherm for the second layer is controlled in order to eliminate hairline cracks.

In order that the present invention can be more readily understood and be put into practical effect, reference will now be made to the accompanying drawings and examples of preferred embodiments of the present invention and wherein:-

Figure 1 is a section through a typical laminate made according to the present invention;

Figure 2 is a pictorial view illustrating a typical moulding method by which the present invention is applied to make a container; and

Figures 3 to 9 are schematic diagrams illustrating stages in a typical method according to the present invention.

Referring to the drawings and initially to Figure 1, there is illustrated a laminated structure 10 comprising an inner layer 11 which is about 4mm thick and an outer layer 12, the inner layer is a filler free vinylester for maximum chemical resistance and the outer layer 12 is about 45mm to 90mm thick providing a supporting backing for the inner layer. In the illustrated embodiment, the outer layer 12 has been deliberately fractured under test conditions at 13 to show how well the inner layer 11 has stayed intimately adhered to the layer 12 in the region of the fracture at 14.

The inner layer 11 has been adhered to the outer layer 12 using the method according to the present invention. For this reason, there is no delamination of the layer 11 from the layer 12.

The layer 11 is preferably made from a thermosetting moulding composition comprising the following:-

Vinylester - 68%;

Continuous glass rovings and/or woven rovings as surface tissues - 30.5%; and

Catalyst - 1.5%.

The layer 12 is preferably made up as follows:-

Resin - 15%;

Glass - 0.5%;

Fine and course river sand - 30%; and

Gravel mix - 54.5%.

In some cases, it may be desirable to apply an outside layer to the layer 12 and this is typically a fire retardant U.V. stable resin which has been coloured.

Application of the present invention to a container is illustrated in Figure 2 where a mould assembly 15 includes an outer body 16 spaced from an internal plug 17. The outer body is mounted on side spring assemblies shown at 18 and a plurality of spaced vibrators 19 are used to vibrate the mould.

In the embodiment of Figure 2, the layer 11 has already been applied to the plug and this has been allowed to harden to a green state and a short time later, the mould is filled as shown at 20 with the composition corresponding to layer 12.

As the mould is being filled, the mould is continuously vibrated so that the sand and gravel particles in the composition 12 act abrasively on the layer 11 to provide an intimate physical interlocking between the layers. Also, as the layer 11 has only partially hardened, there is also a chemical reaction between the two layers which again enhances the adherence between the layers.

In order to reduce the likelihood of hairline fractures in the layer 12, the exotherm of the process occurring during moulding is controlled. In the

illustrated embodiment, for a container measuring about 18 feet in length, 4½ feet high and 4½ feet wide, a pour time prior to gelling of the layer 12 is set at around 1½ hours giving enough time to pour the layer 12 in about the first hour. The vibration is continued for the full 1½ hours and may even be carried on longer than this without adversely effecting the final product.

Figures 3 to 9 illustrate a method for forming the container whereby the method step, illustrated previously in relation to Figure 2, is illustrated in Figures 8 and 9. The method steps illustrated in relation to Figures 3 to 6 are utilised to ease the release of the container from the mould and can be used in conjunction with the method steps of Figures 7 and 8 as previously described. Overall, the method illustrated in relation to Figures 3 to 9 provides a more economical, less time consuming, less labour intensive method for producing a container according to the present invention. Accordingly, this provides economic benefits not available in the prior art.

Figure 3 illustrates schematically a mould 21. The mould is made to the end user's requirements and typically includes inlets, outlets, overflows, fixing inserts and so forth, all of which are prepared in the mould so that these can be integrally moulded. In the case of an electrowinning cell, the mould is formed with recesses and slots to receive FRP panels at a later time. The mould is illustrated as a plain structure in Figure 3, but it will be appreciated that the shape can vary according to the design requirements of the end user.

Figure 4 illustrates schematically a formation of the inner layer. The inner layer comprises a non-filled resin backed with NEXUS cloth, then C.S.M. plus woven rovings, plus C.S.M. at up to a thickness to 4mm to 6mm. In one

typical embodiment surface tissues are employed so that the inner surface closest to the mould has a very high percentage of vinylester resin. This inner layer is formed to maximise chemical resistance and can be provided by any suitable combination of surface membranes, chopped glass rovings, woven glass rovings as reinforcement which is minimised in order to maximise the amount of vinylester resin in the layer. It should be borne in mind that the layer will be later supported by the backing layer, therefore, the inner layer is primarily formed to maximise chemical resistance. Figures 4 and 5 illustrate exemplary stages that can be employed in the preparation of this inner layer.

In Figure 6 there is schematically illustrated release of the inner layer 11 from the mould 21. It will be appreciated that the layer 14 is not, at this stage, at a final cured state. This is because adherence between the layers involves reaction of the outer surface of layer 11 with the backing layer to improve adhesion between the two layers.

The layer 11 which now forms a container is placed over a moulding jig and this is shown in Figure 7. Outer mould walls are located which are fixed or bolted to a main frame and the backing layer is mixed and poured as illustrated in Figures 8 and 9 and is in accordance with the previous description in relation to Figure 2.

When the full backing layer has been applied, gel time is set at one and a half hours and the mould is vibrated for this period of time. Typically cure occurs at four to five hours at 85°C. During the setting time of the backing layer, temperature control is assured by additional hot or cold water by means of a network of galvanised piping located in the outer walls of the mould or in

the jig.

To release the container from the jig and the outer walls, the outer walls are first taken off and then air is applied to lift the completed container off the jig. The container is removed from the jig and stored in heated conditions for twelve hours. The container is then turned over and hand finished around the edges etc. If required, FRP panels are introduced and bonded in place.

It will be appreciated that the backing layer is designed to support the chemically resistant inner layer and can comprise any suitable mixture of resins, glass fibres, glass beads, sands, gravels, timber, sawdust, synthetic fibres, plywood, FRP dust, mineral talcs, earths or cement mix. The selected mixture employed in the method of the present invention will vary according to the needs of the end user.

The present invention therefore provides a reinforced chemically resistant container reliable against leakage and made according to an efficient cost effective method.

The invention has many variations and it will be appreciated that whilst the above has been given by way of illustrative example of the present invention, many variations and modifications thereto will be apparent to those skilled in the art without departing from the broad ambit and scope of the invention as herein set forth.

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:-

1. A container holding acid in an electrolytic cell, the container having a laminate wall comprising an inner layer and an outer layer, the inner layer comprising a composition of vinylester resin, continuous glass rovings in the form of surface tissues providing a very fine membrane of glass having a resin richness greater than 90%, the outer layer comprising vinylester compatible resin to promote bonding with the inner layer, glass fibre, fine, and course sand and gravel.

2. A container according to claim 1 wherein the outer layer comprises by weight 10% to 20% resin, 10% to 20% sand and 30% to 60% gravel.

3. A container according to claim 1 or claim 2 wherein the inner layer is about 4mm thick and the outer layer is from 45mm to 90mm thick.

DATED this 23rd day of December, 1994.

VINYL-CRETE PRODUCTS AUSTRALIA PTY LTD
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ABSTRACT

A laminated structure 10 comprising an inner layer 11 which is about 4mm thick and an outer layer 12, the inner layer is a filler free vinylester for maximum chemical resistance and the outer layer 12 is about 45mm to 90mm thick providing a supporting backing for the inner layer.

A mould assembly 15 includes an outer body 16 spaced from an internal plug 17. The outer body is mounted on side spring assemblies shown at 18 and a plurality of spaced vibrators 19 are used to vibrate the mould the layer 11 is applied to the plug and this is allowed to harden to a green state and a short time later, the mould is filled as shown at 20 with the composition corresponding to layer 12. As the mould is being filled, the mould is continuously vibrated so that the sand and gravel particles in the composition 12 act abrasively on the layer 11 to provide an intimate physical interlocking between the layers.

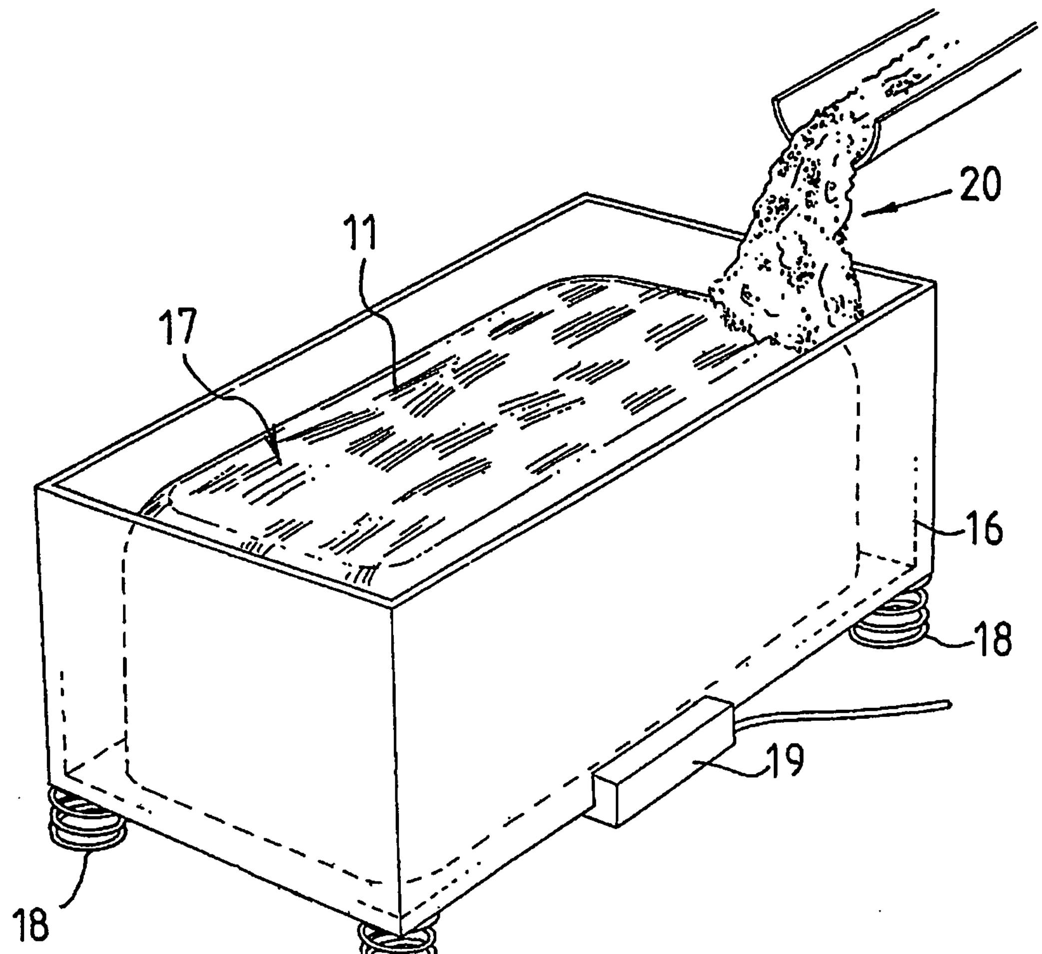

FIG. 2

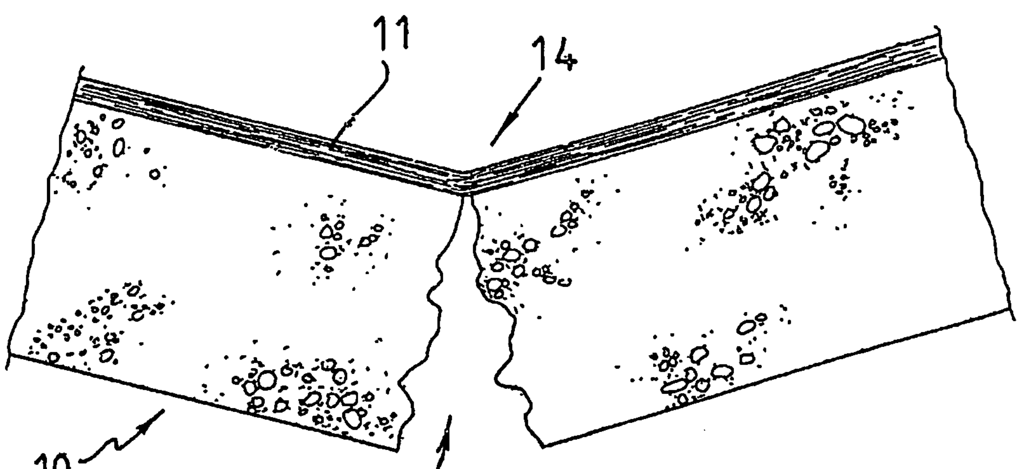

FIG. 1

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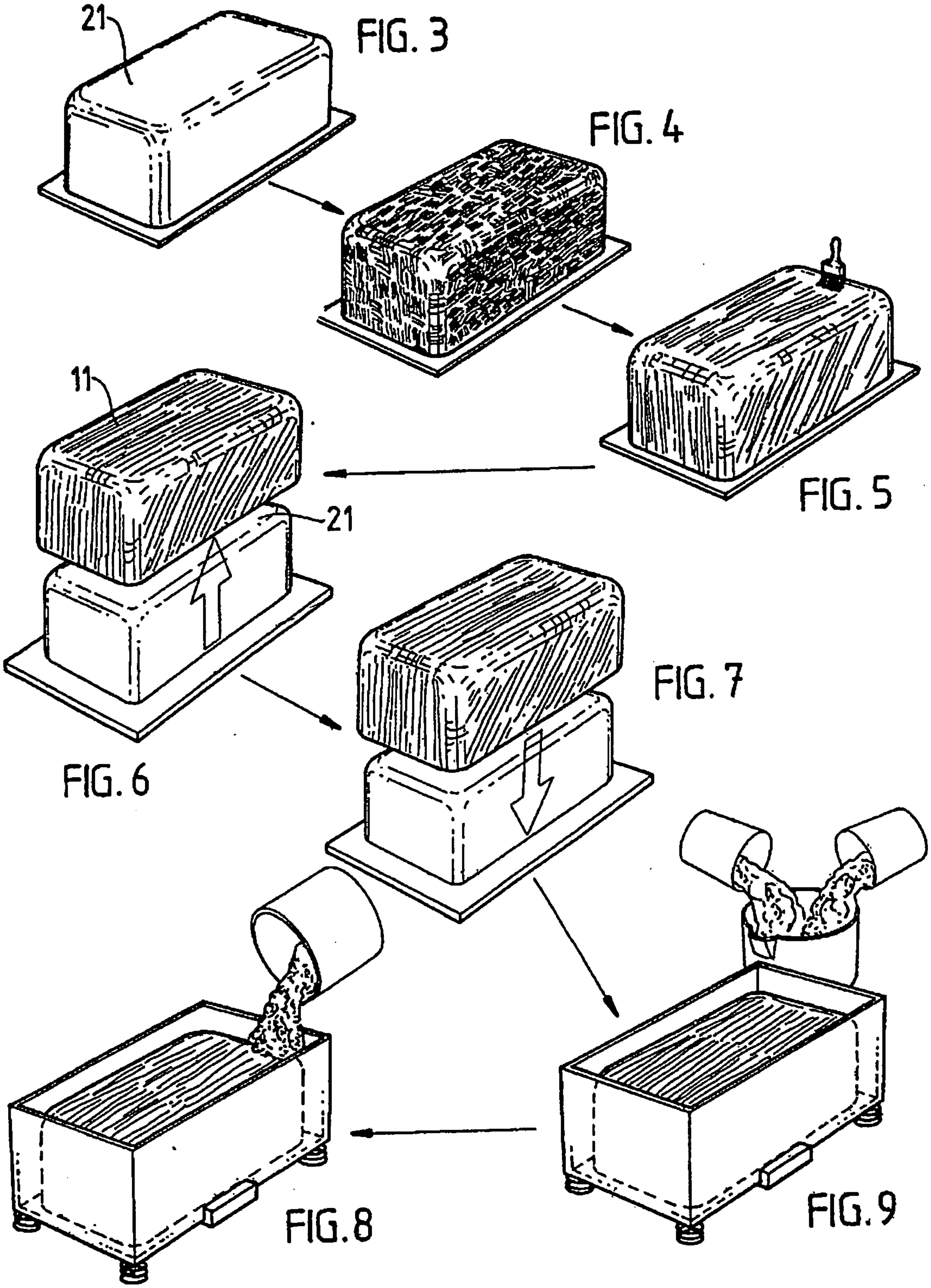

FIG. 3

FIG. 4

FIG. 5

FIG. 6

FIG. 7

FIG. 8

FIG. 9